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EFFECTS OF THERMAL AGING ON A CAST-COMPOSITE PROPELLANT

by

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ABSTRACT. The activation energy, E, frequency factor, A, and specific rate constant, k, were determined for the exposed surface, subsurface, and internal regions of five cast-composite propellant (ANP-2639 AF) motors thermally aged at various temperatures from 95—165°F for different periods. The E values of the exposed surfaces for the 95 and 130°F aged samples are essentially identical (23 kcal/mole), while there is a 4 kcal/mole increase for the samples aged at 150 and 165°F. For the subsurface regions, 0.005—0.010 inch below the exposed surface, and for the internal region, 0.5 inch below the exposed surface, the E values show good similarity—each is approximately 25 kcal/mole. The study shows also that E, as well as A, vary with the aging history of the sample.

A comparison of all E values and a study of the relationships of temperature to a rate factor τ/T^2, indicate that for the thermally aged propellant grains, the primary component altered at the surface is the binder, whereas, in the interior, it is the oxidizer.

U.S. NAVAL ORDNANCE TEST STATION

China Lake, California

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U. S. NAVAL ORDNANCE TEST STATION

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FOREWORD

A propellant evaluation program for predicting safe-life was begun on 1 June 1961 and is being conducted at the U. S. Naval Ordnance Test Station. The work covers the determination of physical and chemical properties of propellants to predict the critical temperature of a given size and configuration of propellant, and the time to cook-off if this critical temperature is exceeded.

This report summarizes the results of the investigations of the reaction kinetics for thermally aged cast-composite propellant samples and describes the usefulness of microimmersion autocatalytic analysis (MAA) as a rapid, reliable method for the acquisition of reaction kinetic data (E, A, and k values) from small-scale tests.

The work has been performed under Task Assignment RMMP-21-001/216-1/F009-01-016. This report has been reviewed for technical accuracy by Colin A. Taylor, Head, Product Evaluation Branch.

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INTRODUCTION

Late in Fiscal Year 1959, a project was inaugurated at the U. S. Naval Ordnance Test Station (NOTS) to study the effect of aging on the ignitibility of a cast-composite propellant, ANP-2639 AF[1]. During Fiscal Year 1960, the new casting facility at NOTS was in operation, and twenty 8-pound test motors were cast. Two types of perforation were used: 10 motors were prepared with the "wagon-wheel" perforation, and 10 with an eight-point-star perforation. The motors were thermally aged at various temperatures from 95–165°F for different time periods. Since undergoing the aging treatment, the motors had been stored at 70°F. An examination of the aluminum foil end-seals on the motors revealed broken seals on all motors. Seventeen were broken at both ends and three at only one end. Although the majority of the openings were small and cracklike, it was assumed that the interior portions of all the grains were subjected to the atmospheric conditions within the 70°F oven. The final condition of the exposed propellant surface is the result of atmospheric oxidation, including any possible moisture effect plus the initial effect of the mandrel, with its off-release compound, at the time of casting the grain. Although there may have been a moisture effect, real effects of aging at different temperatures were detected.

The five motors chosen from the original 20 for this study had the wagon-wheel perforation and included one from each of the following aging treatment groups:

Aging temp., °F	Time, days
95	120
130	10
130	120
150	30
165	10

A microimmersion autocatalytic analysis (MAA) method has been evolved for the rapid determination of the activation energy, E, and frequency factor, A, of propellants. In this method, a very small sample of propellant (5–10 mg) is brought immediately to temperature by dropping it in a constant-temperature, inert oil bath[2], and the time-to-deflagration, τ, is recorded for various temperatures, T. E is then determined in a

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[1] The composition for ANP-2639 AF propellant is given in the Propellant Powder Manual, SPIA/M2.

[2] This microimmersion technique reduces the induction period, because of the better thermal conductivity of oil as compared to air, and thus simplifies and reduces the amount of experimental data required.

manner similar to that used by Galwey and Jacobs (Ref. 1) for their method of dropping a sample into an empty glass tube embedded in a heavy copper heating block within a furnace. This consists of plotting log τ/T^2 versus the reciprocal of the Kelvin temperature of the heating block and determining E from the slope of this line (Fig. 1). It has been demonstrated (Ref. 2, Table 3) that there is reasonable agreement in the values of E, as determined by MAA and by other methods, such as differential thermal analysis (DTA), derivative differential thermal analysis (DDTA), and the Dewar-flask onset method (DOM). The agreement ranges over a mass change from 5–10 mg for the MAA method, 20–30 mg for DTA, 50–150 mg for DDTA, and up to 20 grams for DOM.

The method used in this study to determine A is based on the equation derived by Galwey and Jacobs (Ref. 1):

$$\frac{1}{\tau} = \frac{QAE}{cRT^2} e^{-E/RT}$$

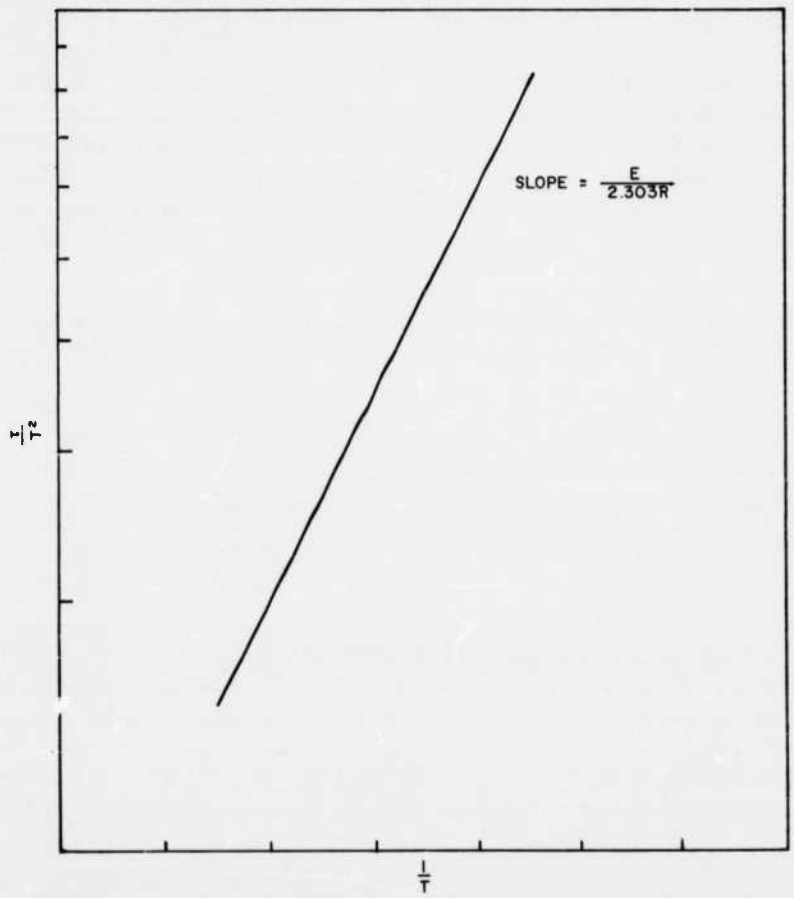


FIG. 1. Typical Graph for the Relationship of Time to Deflagration Versus Temperature for the Evaluation of E as Used in MAA Method.

This equation is rearranged to give

$$\frac{cR}{QE} \cdot \frac{T^2}{\tau} = Ae^{-E/RT} = k$$

where T is the Kelvin temperature, c the heat capacity of the reactant, R the standard gas constant, Q the heat of reaction, E the energy of activation, and A exp(-E/RT) the specific rate constant of the reaction. The specific rate constant k is the familiar Arrhenius equation that has been utilized for the determination of k values at various temperature levels.

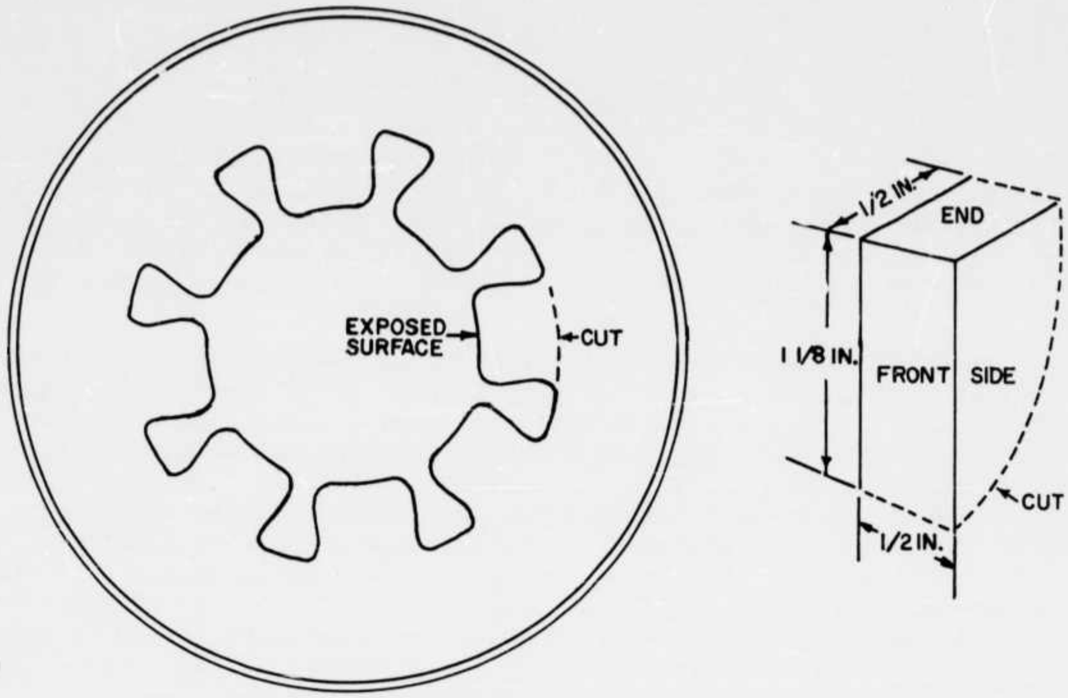
The reported values for E, as determined by MAA and DTA, agree within 1 kcal/mole on unaged ANP-2639 AF propellant. The difference in the reported values for A and k as determined by these methods probably was due to the different conditions of analyzing. The DTA data are obtained at the first exothermic peak under a given heating rate, while the MAA data, taken at a constant temperature, are generally taken at approximately 100°F below the DTA peak. Data obtained at lower temperatures could effect the A and k values, and this contention is supported further by Anderson and Freeman (Ref. 3). The actual conditions of analysis between DTA and MAA are different. In MAA, the propellant sample is surrounded by oil; in DTA, glass beads of approximately 0.1 mm diameter are used. This difference in thermal conduction methods could contribute to a difference in results.

Although the MAA technique is not as comprehensive as DTA, its multiple advantages, such as speed and ease and simplicity of operation, make it a valuable tool applicable to applied research studies. Considerable information may be obtained easily within a short time. The acquisition of a new high temperature furnace and the availability of a high temperature, liquid heat-transfer medium at NOTS has made it possible to study the reaction kinetics of cast composite propellants by this technique.

In this study, the reaction kinetics for a cast composite propellant (ANP-2639 AF), thermally aged at various temperatures from 95–165°F for different time periods, were investigated. Three regions of the grain were studied: the exposed surface, the subsurface, which is just below the 0.005- to 0.010-inch skin of the exposed surface, and the internal portion, which is 0.5 inch below the exposed surface.

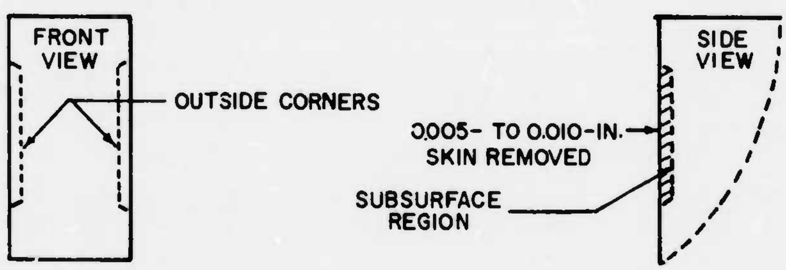
SAMPLE PREPARATION

A wedge was cut from one lug of each propellant grain (Fig. 2), from which 5- to 10-mg samples were obtained from three different regions (Fig. 3). Samples for the exposed surface region were taken from the outside corners of the lug, which had been exposed to the atmospheric conditions within the perforation of the grain (Fig. 3a). The samples for the subsurface region were taken from a section of the propellant

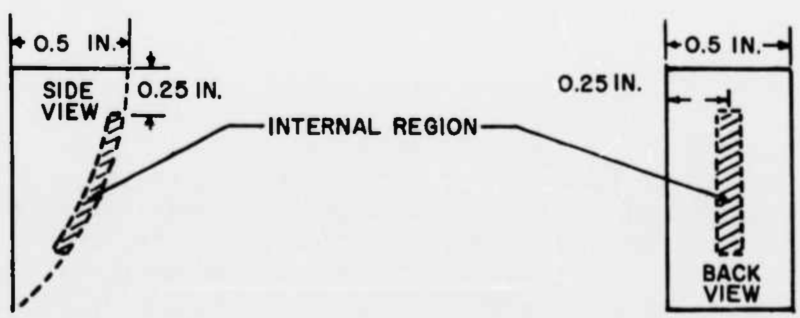


a. End View of Propellant Grain. b. Wedge.

FIG. 2. Propellant Grain and Sample Wedge.



a. Exposed surface region. b. Subsurface region.



c. Internal region.

FIG. 3. Sample Preparation From Wedge.

0.005–0.010 inch below the skin on the exposed side of the lug (Fig. 3b). Test samples for the internal region were taken from the inside center portion of the wedge (Fig. 3c). The wedges were uniform in size, and the 5- to 10-mg test samples from each region were prepared just before making the determinations.

EQUIPMENT AND INSTRUMENTATION

The equipment and instrumentation for the tests consisted of a heating block, thermocouple probe, sample tube, illuminating light, funnel, magnifying glass, sample-dropping device, liquid heat-transfer medium, and an indicating-controlling pyrometer (Fig. 4).

FIG. 4. Equipment and Instrumentation Used in MAA Tests.

The external surface of the cylindrical aluminum heating block is non-inductively wound with B & S 25-gage Nichrome wire. The upper portion of the block has a vertical cylindrical hole of sufficient size to accommodate securely a 10- by 75-mm pyrex test tube. A horizontally placed

3/8-inch sight hole extending through the heating block and perpendicular to the sample tube hole is positioned so that the operator can observe the bottom portion of the test tube. This bottom portion is illuminated by placing a light outside the rear sight hole. The temperature of the tube is measured by means of a B & S 28-gage chromel-alumel thermocouple placed in the block within 2-3 mm of the tube. Although there may be some slight difference between the temperature of the thermocouple and that of the test tube, this difference, which has been determined to be a maximum of 2°F in the range studied, would be constant through the temperature range employed, and any difference would be within experimental error. The entire assembly is housed within an insulated gallon-size container.

The speed and accuracy of the MAA determinations depend, to a large measure, on the precision at which a constant temperature can be maintained. During these tests, constant temperatures were maintained by using a Wheelco Model 402 Truline Capacitrol[3] indicating-controlling pyrometer, which contains a proportioning-controlling system for maintaining constant temperatures.

A polyphenyl ether, OS-124[4], was used as the high temperature heat-transfer medium. The fluid performed satisfactorily throughout the period of investigation, and the reaction times at various temperature levels were reproducible. However, 1½ weeks after completing the test series, the color of the fluid changed from a very faint yellow to a darker straw color, and some difficulty was experienced in obtaining reproducible reaction times. It is suggested, therefore, that a fresh supply of the fluid be employed at the beginning of a test series, and that the storage bottle and the test tubes containing the fluid should always be shielded against the effects of direct and indirect sunlight.

PROCEDURE

The heating block was brought to the desired temperature, and the test tube, containing 0.7-1.0 ml of the fluid, was inserted into the well and given a dwell time of 8-10 minutes to allow the temperature of the fluid to become constant. The propellant sample then was dropped remotely by a control arm into the reaction vessel (test tube).

The propellant sample must come to rest at the bottom-center of the tube. If it rests at the bottom-side of the tube, deflagration (rapid bubbling) may occur, but the sample may not rise to the surface of the fluid. It is recommended also that the samples be cubical or rectangular

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[3] A product of Barber-Colman Company, Wheelco Industrial Instruments Division.

[4] A product of Monsanto Chemical Co., Organic Chemicals Division.

in shape, as thin, flaky samples have a tendency to remain on the surface of the liquid. The time required for the samples to come to a resting position at the bottom of the tube should be constant. During these tests, time was recorded when the propellant sample came to rest on the bottom of the tube and again when it deflagrated. The moment of deflagration was fixed when the sample rose toward the surface and passed up beyond the line of sight of the observation window. Two or three determinations were run for each temperature level. A clean tube and new fluid were used for each test.

At the completion of each test, while the tube was still warm, the contents were discarded into an appropriate container. After cooling, the tube was soaked in xylene for a short period, cleaned with a brush, rinsed with acetone, and then inverted for drying.

RESULTS AND DISCUSSION

E, A, and k values were determined for three regions of five cast-composite propellant (ANP-2639 AF) motors, thermally aged at various temperatures from 95–165°F for different time periods. The parameters E and A are two of the many required for the determination of the critical temperature and the time-to-explosion or deflagration of propellants (Ref. 2). The k values were used to verify the E values obtained with the method used by Galwey and Jacobs. The relationship of temperature versus the rate factor, τ/T^2, was examined in the study of the temperature profiles at three regions of the thermally aged propellant. The temperature versus time-to-deflagration data were obtained by the MAA method, and were programmed for computation on the IBM 7090 computer to calculate E, A, and k values.

Before starting the tests on the thermally aged ANP-2639 AF propellant samples, the MAA technique was checked on a fresh sample of ANP-2639 AF, and an E value of 26.8 kcal/mole was determined. This compared favorably with the 27.8 kcal/mole value obtained by DTA on a similar fresh sample. The precision of the determinations can possibly be affected by the inherent physical characteristics of the propellant sample. Because of the small sample size (5–10 mg), the distribution of the ingredients may not be homogeneous; therefore, the sample may contain a minimum of four combinations:

1. Theoretical percentage of ammonium perchlorate, aluminum, and binder
2. Predominantly ammonium perchlorate and binder
3. Predominantly aluminum and binder
4. Predominantly binder

The activation energies of the exposed surfaces for the 95 and 130°F aged samples are essentially identical (23 kcal/mole), while the E values for the 150 and 165°F samples were 27 kcal/mole (Fig. 5). This 4 kcal/mole <u>increase</u> for the samples aged at 150 and 165°F could indicate that

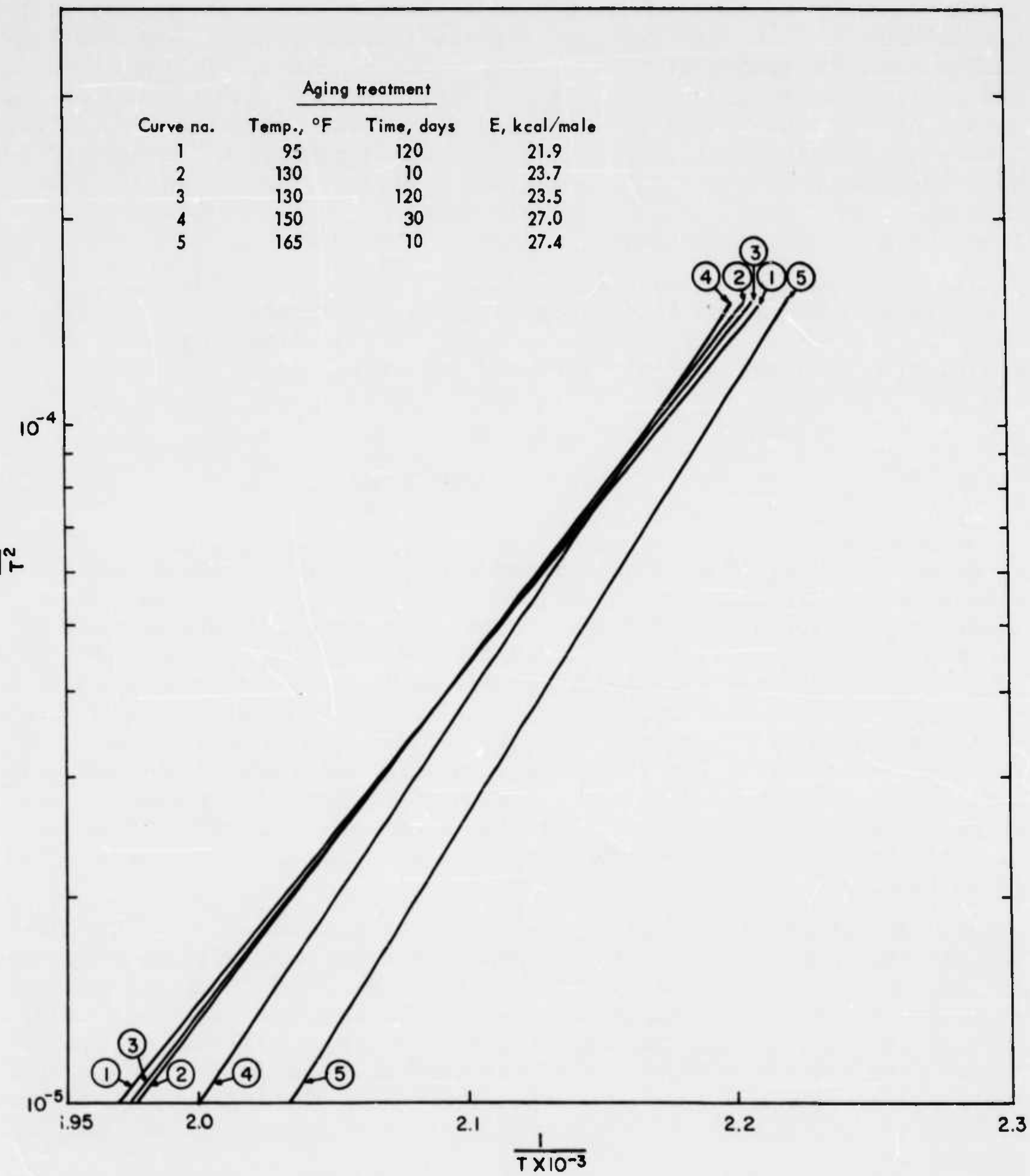

FIG. 5. Comparison of E Values for Thermally Aged ANP-2639 AF Propellant (Exposed Surface).

temperatures greater than 130°F cause a degradation of the binder portion of the exposed surface, as evidenced by a hardening of the surface region. Such action (atmospheric oxidation) possibly is one reason for the increased difficulty experienced in igniting aged propellant grains. Curves 4 and 5 (for the 150 and 165°F samples) of Fig. 5 are displaced progressively along the axis of abscissas toward the lower temperature values

of the graph. For the exposed surfaces, A, as well as E, increases for propellant samples aged above 130°F (Table 1). This large increase in A indicates that the exposed surface becomes thermally less stable under the conditions studied.

TABLE 1. Values for the Activation Energy, E, Frequency Factor, A, and Specific Rate Constant, k, at 360°F for Thermally Aged ANP-2639 AF Propellant

Aging treatment		E,	A	$k_{360°F}$
Temp., °F	Time, days	kcal/mole	(sec^{-1})	(sec^{-1})
A. Exposed Surface				
165	10	27.4	2.5×10^9	1.7×10^{-4}
150	30	27.0	1.2×10^9	1.2×10^{-4}
130	120	23.5	2.8×10^7	1.6×10^{-4}
130	10	23.7	3.6×10^7	1.5×10^{-4}
95	120	21.9	6.1×10^6	1.9×10^{-4}
B. Subsurface (0.005–0.010 Inch Below Surface)				
165	10	24.3	1.3×10^8	2.9×10^{-4}
150	30	25.8	4.3×10^8	1.7×10^{-4}
130	120	24.8	1.4×10^8	1.8×10^{-4}
130	10	26.1	4.6×10^8	1.3×10^{-4}
95	120	24.5	9.1×10^7	1.6×10^{-4}
C. Internal (0.5 Inch Below Surface)				
165	10	24.3	1.5×10^8	3.2×10^{-4}
150	30	25.6	4.5×10^8	2.2×10^{-4}
130	120	24.8	1.4×10^8	1.8×10^{-4}
130	10	25.4	2.3×10^8	1.5×10^{-4}
95	120	25.9	3.3×10^8	1.2×10^{-4}
Fresh sample (MAA)		26.8	9.6×10^8	1.3×10^{-4}*
Fresh sample (DTA)		27.8	1.5×10^8	6.9×10^{-6}*

* See text for explanation of the difference in values obtained from MAA and DTA.

The values of E for the subsurface regions (0.005–0.010 inch below the exposed surface) for all samples, regardless of temperature, are essentially identical, 25 kcal/mole (Fig. 6). Curves 1 and 2 of Fig. 6 are very similar, as are curves 3 and 4. Curve 5 again is displaced along the axis of abscissas toward the lower temperature values of the graph. This again

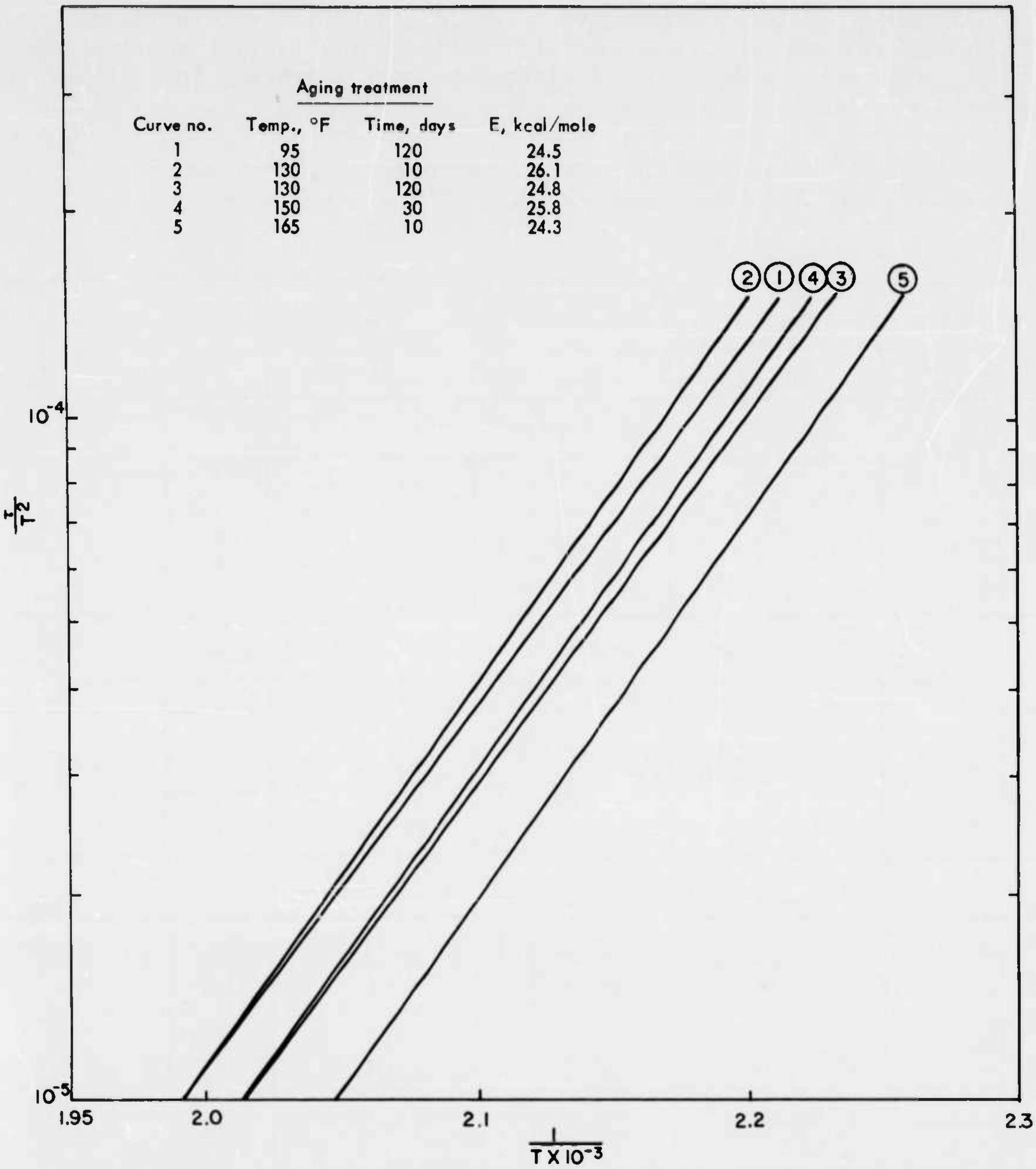

FIG. 6. Comparison of E Values for Thermally Aged ANP-2639 AF Propellant (Subsurface, 0.005–0.010 Inch Below Exposed Surface).

indicates that changes in the reaction kinetics in the subsurface regions, though not always linear, can be induced by subjecting this propellant to high temperature storage (Table 1).

The E values for the internal regions, 0.5 inch below the exposed surface, are essentially equal for all samples—approximately 25 kcal/mole. The curves are parallel to each other and are displaced progres-

sively along the axis of abscissas (Fig. 7). This indicates that A varies with the aging history of the sample, a slight decrease when the temperature increases from 95 to 130°F and a general leveling off above 130°F. (In general, the greater effect is toward the surface of the propellant.)

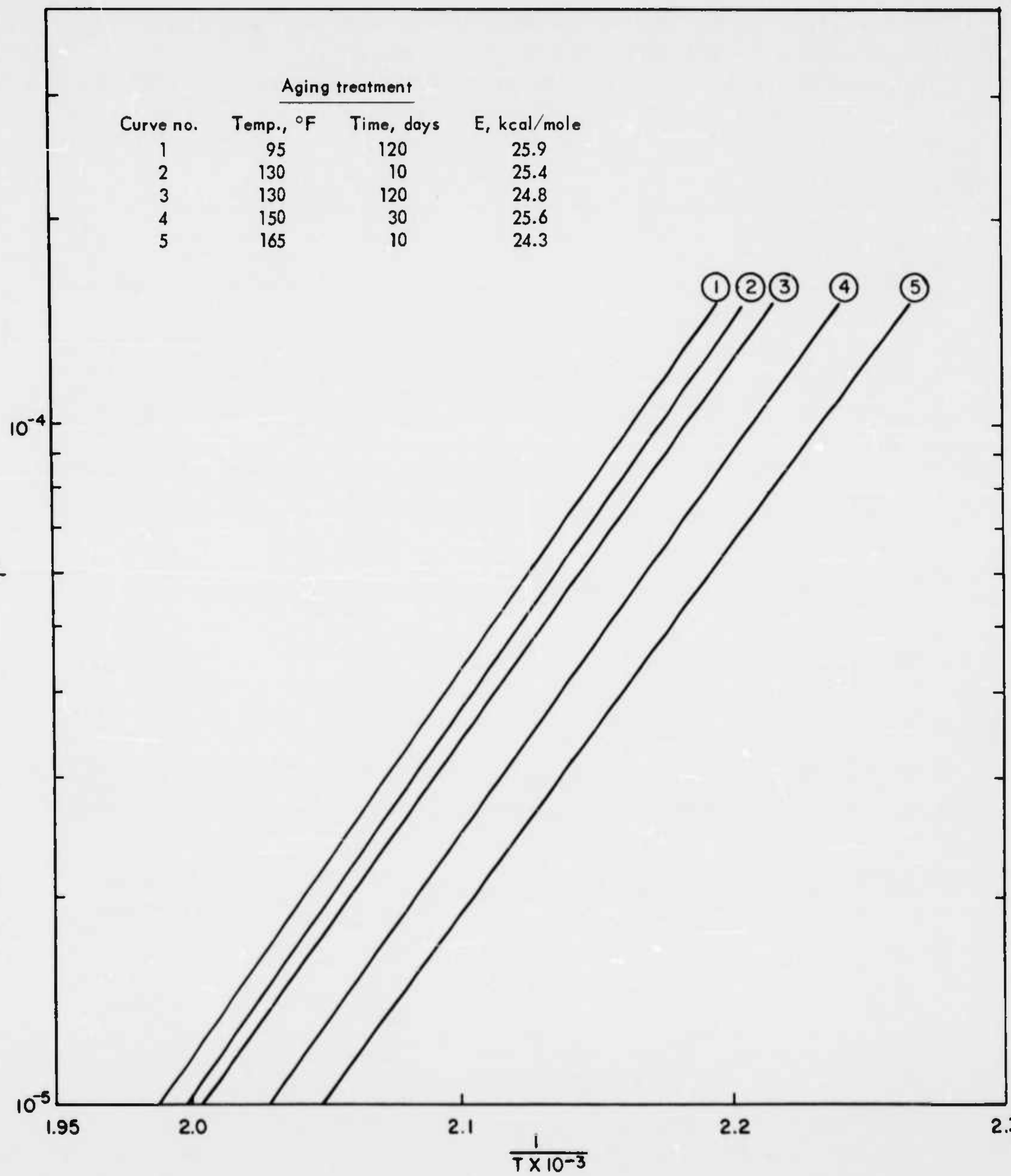

FIG. 7. Comparison of E Values for Thermally Aged ANP-2639 AF Propellant (Internal, 0.5 Inch Below Exposed Surface).

The values for the specific rate constant, k(sec^{-1}), at various temperature levels for thermally aged ANP-2639 AF propellant are given in Table 2. An Arrhenius plot for several temperature levels of log k(sec^{-1}) versus the reciprocal of the Kelvin temperature gave good checks (within 0.1 kcal/mole) for E values as compared with those obtained by the method shown in Fig. 1 and with the IBM 7090 computer.

A comparison of all E values for the subsurface and inside regions shows good similarity; the maximum difference is 1.8 kcal/mole (Fig. 8). However, there is a marked difference between the subsurface and the exposed surface region; the maximum difference is 3.1 kcal/mole. Figure 8

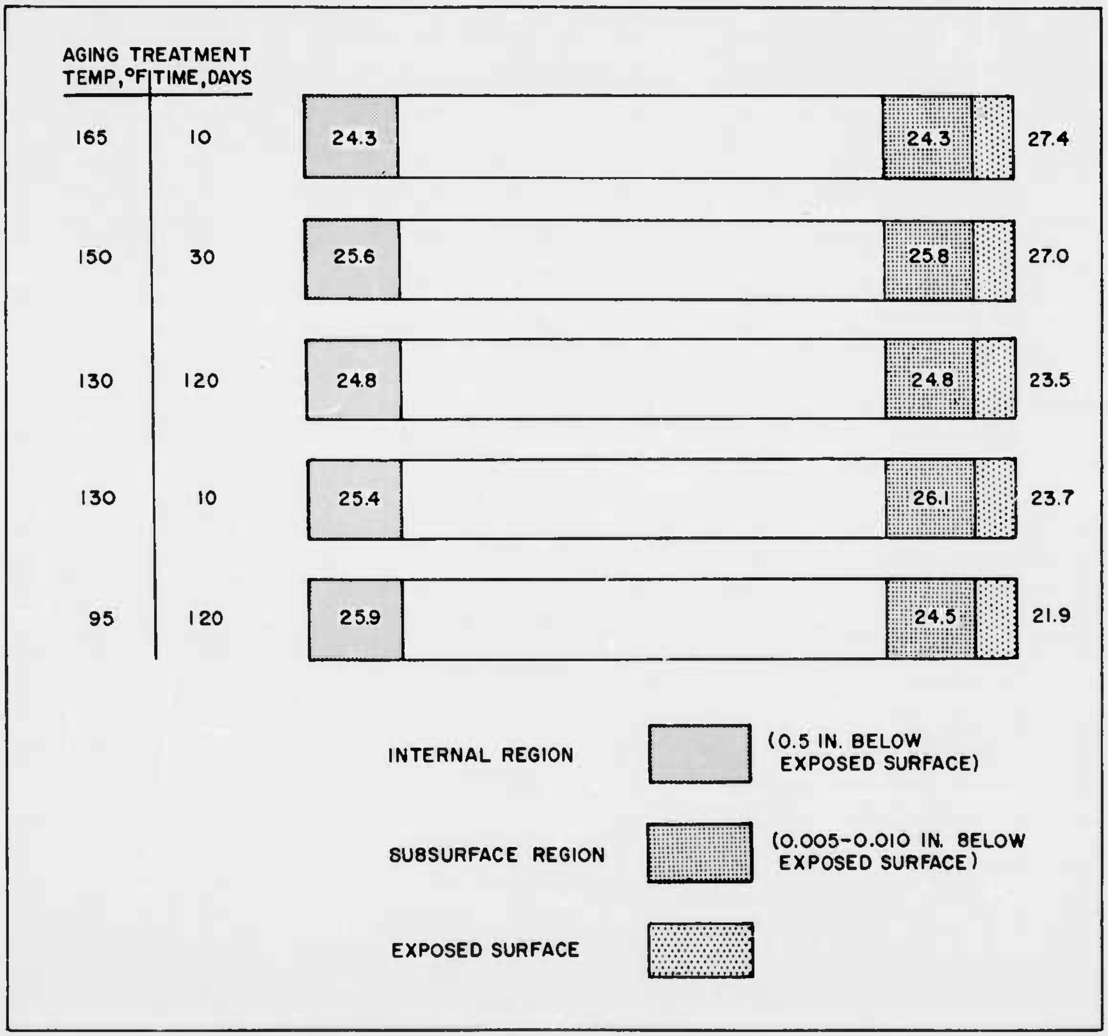

FIG. 8. Comparison of E Values (in kcal/mole) at Various Regions for Thermally Aged ANP-2639 AF Propellant.

TABLE 2. Values for the Specific Rate Constant, k(sec^{-1}), at Various Temperature Levels for Thermally Aged ANP-2639 AF Propellant

Aging treatment		k(sec^{-1})							
Temp., °F	Time, days	350°F	360°F	370°F	380°F	390°F	400°F	410°F	420°F
		A. Exposed Surface							
165	10	1.2 x 10^{-4}	1.7	2.5	3.6	5.0	7.1	9.9	1.4 x 10^{-3}
150	30	9.5 x 10^{-5}	1.2	1.8	2.5	3.5	4.9	6.9	9.1 x 10^{-4}
130	120	1.1 x 10^{-4}	1.6 } x 10^{-4}	2.1 } x 10^{-4}	2.9 } x 10^{-4}	3.9 } x 10^{-4}	5.2 } x 10^{-4}	6.9 } x 10^{-4}	9.1 x 10^{-4}
130	10	1.1 x 10^{-4}	1.5	2.0	2.8	3.7	5.0	6.7	8.9 x 10^{-4}
95	120	1.4 x 10^{-4}	1.9	2.6	3.5	4.6	6.0	7.8	1.0 x 10^{-3}
		B. Subsurface (0.005–0.010 Inch Below Surface)							
165	10	2.1 x 10^{-4}	2.9	4.1	5.6	7.6	1.0 x 10^{-3}	1.4 x 10^{-3}	1.8 x 10^{-3}
150	30	1.2 x 10^{-4}	1.7	2.4	3.4	4.7	6.5 x 10^{-4}	8.9 x 10^{-4}	1.2 x 10^{-3}
130	120	1.3 x 10^{-4}	1.8 } x 10^{-4}	2.6 } x 10^{-4}	3.5 } x 10^{-4}	4.9 } x 10^{-4}	6.6 x 10^{-4}	8.9 x 10^{-4}	1.2 x 10^{-3}
130	10	9.2 x 10^{-5}	1.3	1.9	2.6	3.6	5.0 x 10^{-4}	6.9 x 10^{-4}	9.4 x 10^{-4}
95	120	1.0 x 10^{-4}	1.6	2.5	3.4	4.5	6.0 x 10^{-4}	7.9 x 10^{-4}	9.9 x 10^{-4}
		C. Internal (0.5 Inch Below Surface)							
165	10	2.4 x 10^{-4}	3.2	4.4	6.0	8.2	1.1 x 10^{-3}	1.5 x 10^{-3}	1.8 x 10^{-3}
150	30	1.7 x 10^{-4}	2.2	3.1	4.3	6.0	8.2 x 10^{-4}	1.1 x 10^{-3}	1.4 x 10^{-3}
130	120	1.2 x 10^{-4}	1.8 } x 10^{-4}	2.5 } x 10^{-4}	3.4 } x 10^{-4}	4.8 } x 10^{-4}	6.4 x 10^{-4}	8.7 x 10^{-4}	1.2 x 10^{-3}
130	10	1.0 x 10^{-4}	1.5	2.1	2.9	4.0	5.4 x 10^{-4}	7.4 x 10^{-4}	1.0 x 10^{-3}
95	120	9.6 x 10^{-5}	1.2	1.8	2.5	3.4	4.7 x 10^{-4}	6.4 x 10^{-4}	8.2 x 10^{-4}
Fresh sample		9.2 x 10^{-5}	1.3 x 10^{-4}	1.9 x 10^{-4}	2.7 x 10^{-4}	3.8 x 10^{-4}	5.3 x 10^{-4}	7.3 x 10^{-4}	1.0 x 10^{-3}

also shows that there is a positive Δ E between the subsurface and surface layers at the two higher temperature levels, while there is a negative Δ E at and below the aging temperature level of 130°F for 120 days. This would indicate that there are two possible mechanisms involved in the oxidation of the propellant surface. The 95 and 130°F levels possibly could be represented by atmospheric oxidation. After aging at the 150 and 165°F levels, the activation energy is on an increase, which would indicate a different oxidation path, such as an ammonium perchlorate-atmospheric oxidation combination.

A study of the relationships of temperature to a rate factor τ/T^2 of the thermally aged propellant samples (Fig. 9) indicates that there

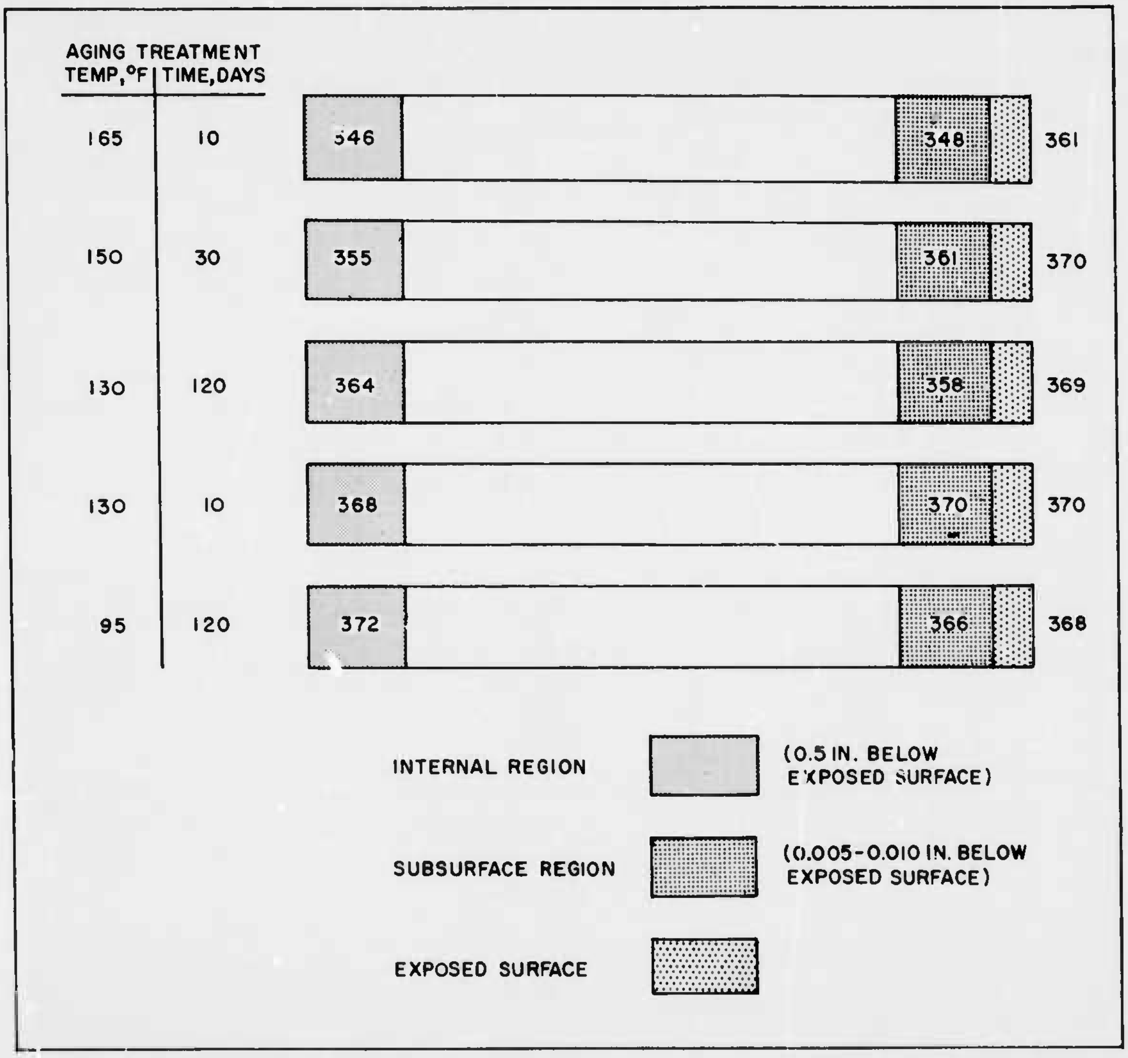

FIG. 9. Relationship of Temperatures to Rate Factor τ/T^2 at $\tau/T^2 \times 10^{-4}$ for Thermally Aged ANP-2639 AF Propellant. All temperatures are °F.

are pronounced discontinuities in the temperature profiles at the two surface regions while there is a continuous progressive change at the inside region. The subsurface region shows an appreciable difference above the aging levels of 130°F for 10 days and 150°F for 30 days, while the exposed surface region shows a significant difference only above the 150°F level (Fig. 10). This indicates that there are mixed oxidation reactions occurring at these levels and that at higher temperature levels (above 150°F) the reactions are able to overcome the hard, smooth surface layer formed by atmospheric oxidation.

In the schematic representation of the oxidation processes occurring in thermally aged ANP-2639 AF propellant (Fig. 11), the area parameters are not absolute boundaries, and the differently marked areas indicate only the predominate mode of action present in each region. For thermally aged propellant grains, the primary component altered at the surface is the binder, whereas, in the interior, it is the oxidizer.

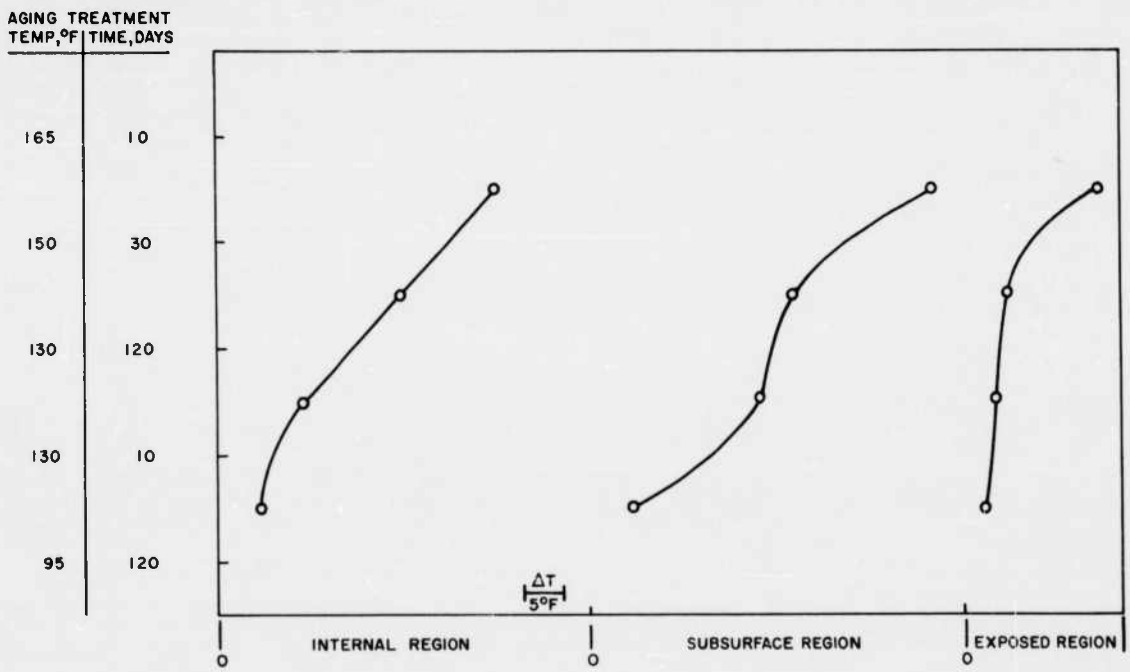

FIG. 10. Cumulative Temperature Differences at $\tau/T^2 \times 10^{-4}$ for Thermally Aged ANP-2639 AF Propellant.

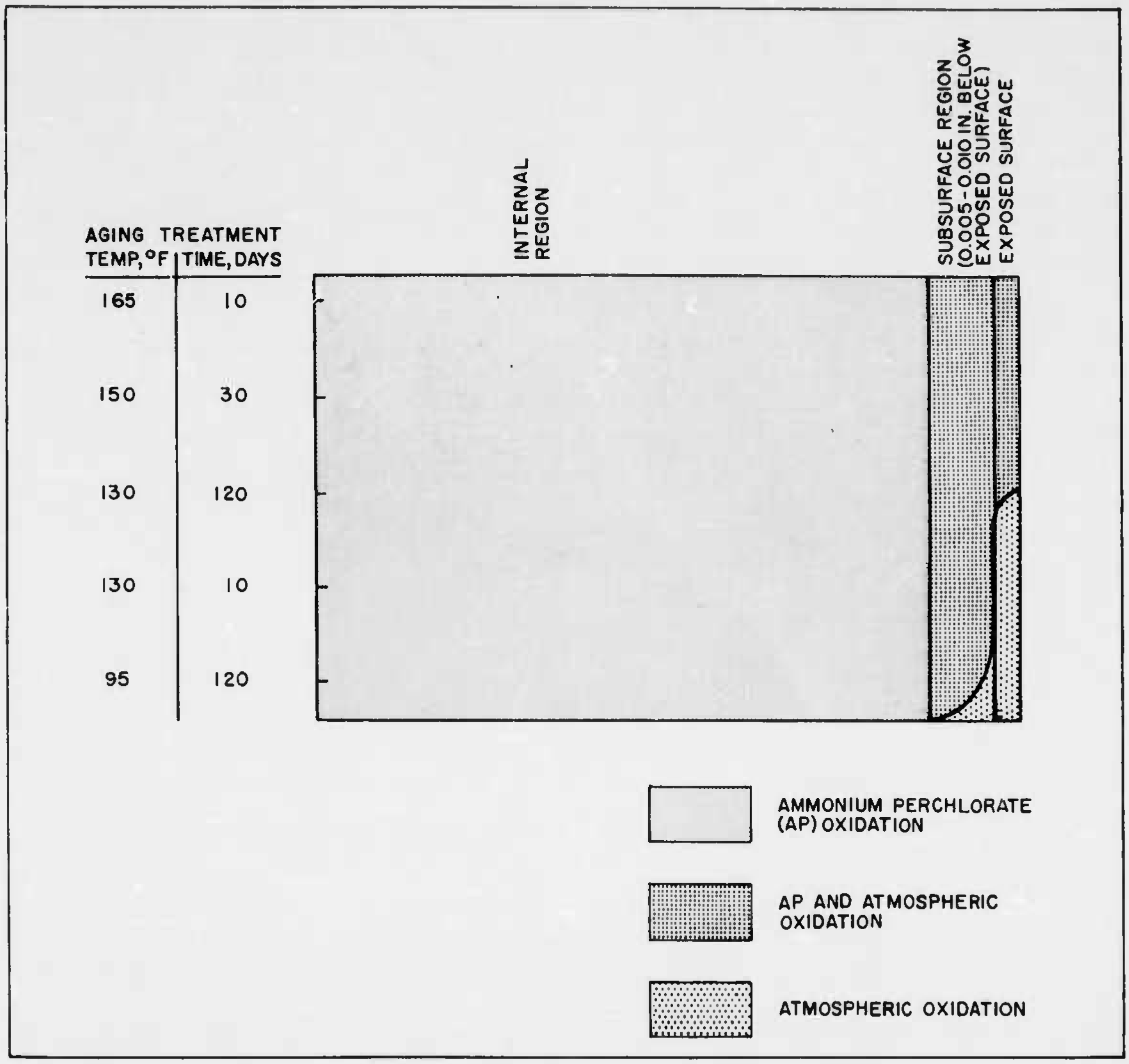

FIG. 11. Graphic Representation of Oxidation Process for Thermally Aged ANP-2639 AF Propellant.

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